COMPARISON OF SHEAR BOND STRENGTH OF VENEERING CERAMIC TO ZIRCONIA AFTER SURFACE TREATMENT WITH AIR ABRASION AND LASER TREATMENT– AN IN VITRO STUDY.

Dissertation submitted to

The Tamil Nadu Dr.M.G.R. Medical University

In partial fulfilment of the degree of

MASTER OF DENTAL SURGERY

BRANCH I

PROSTHODONTICS AND CROWN & BRIDGE

2015-2018

CERTIFICATE

This is to certify that the dissertation entitled **"Comparison of shear bond strength of veneering ceramic to zirconia after surface treatment with air abrasion and laser treatment- An In vitro study."** is a bonafide record of the work done by Dr.Ebinu. A Post graduate student during the period 2015-2018 under my guidance and supervision. This dissertation is submitted in partial fulfilment of the requirements for the award of MASTER OF DENTAL SURGERY IN BRANCH I (PROSTHODONTICS AND CROWN & BRIDGE) under THE TAMIL NADU Dr. M.G.R MEDICAL UNIVERSITY, GUINDY, CHENNAI. It has not been submitted (partial or full) for the award of any other degree or diploma.

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"Enlightment is just another word for feeling comfortable with being a completely ordinary person."

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CONTENTS IN CONCISE

CONTENTS

LIST OF ABBREVIATIONS

ANOVA	Analysis of Variance
Al_2O_3	Alumina
CAD	Computer Aided Designing
CAM	Computer Aided Manufacturing
CTE	Coefficient of Thermal Expansion
EDS	Energy Dispersive X-ray Spectroscopy
ESEM	Environmental Scanning Electron Microscopy
MPa	Mega Pascal
Nd YAG	Neodymium-doped Yttrium Aluminium Garnet
SEM	Scanning Electron Microscope
SBS	Shear Bond Strength
YSZ	Yttria Stabilized Zirconia
Y-TZP	Yttria stabilized Tetragonal Zirconia Polycrystals

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ABSTRACT

INTRODUCTION:

The use of All Ceramic restorations as an alternative to metal ceramic restorations has substantially increased over the last few decades. Zirconia serves as a core ceramic in fixed dental restoration as it has excellent biocompatibility, wear resistance, flexural strength, and fracture toughness. However, delamination and chipping of veneer are the two common failure modes of zirconia restorations.

The success of zirconia based all ceramic restorations dependent on the stable bonding between zirconia core and veneering ceramic. Enhancement of the adhesion between the zirconia substrate and the veneering ceramic is essential for the clinical success of zirconia restorations. The present study investigated the effect of various surface treatments on the interfacial bonding between zirconia and ceramic.

Here, the purpose of the present study is to evaluate and compare the shear bond strength between zirconia and veneering ceramic after surface treatment with sandblasting and laser.

AIMS AND OBJECTIVES:

1. Determination of shear bond strength between zirconia and veneering ceramic after sandblasting with 110-micron Alumina (Al_2O_3) particles.
2. Determination of shear bond strength between zirconia and veneering ceramic after Nd YAG laser treatment.
3. Comparison of shear bond strength of veneering ceramic to zirconia
   i. without surface treatment,
   ii. with sandblasting and
   iii. with laser treatment.

METHODOLOGY:

In the present study, specific dimension of zirconia block was designed using Design software. The file created was saved in STL format. As per the data available 3Y-TZP block was machined to 7 mm thickness, 7 mm breadth and 15 mm length rectangular samples using milling machine. These samples were sintered using sintering furnace.

These samples were cleaned ultrasonically using Ultrasonic cleaner and further cleaned with demineralized water, followed by drying in hot air. Among thirty zirconia blocks ten zirconia were sandblasted, ten were laser treated and remaining were taken as control.

All specimens were veneered with ceramic and subjected to shear stress in Instron 3345 Universal testing machine. The data were analysed with ANOVA followed by Dunnett ‘t’ test with a significance value less than 0.05 ($p<0.05$).

Scanning Electron Microscopy of the sandblasted and laser treated samples were done at 500X, 1000X, 2000X to evaluate the surface roughness of the zirconia surface after surface treatments.

RESULTS

In the present study the mean value of shear bond strength was 28.30±2.92 MPa for control group, 33.92±2.67 MPa for sandblasted and 41.67±5.16 MPa for laser treated group. These results were statistically analyzed using ANOVA (Post hoc) followed by Dunnett ‘t’ test applied and find that these results were statistically significant ($p<0.05$) between the groups.

SUMMARY AND CONCLUSION

Although it was proved that surface treatment improved the bond strength of both sandblasted and laser treated zirconia; the increase in bond strength of laser treated specimen was higher than that of sandblasted one. However further clinical research is suggested in order to prove it as a reliable and successful modality.

INTRODUCTION

Metal ceramic restorations were available for three decades. This type of restorations had gained popularity because of its favourable performance and good esthetics. Despite its success, the demand for improved aesthetics and the concerns regarding the biocompatibility of the metal has lead to the introduction of all-ceramic restorations .[1,2,3]

All ceramic restorations use ceramic materials such as feldspathic, glass–ceramic, and glass-infiltrated ceramic types. Recently, alumina and zirconia were added to the list.[4,5] Silica-based ceramics, such as feldspathic porcelain, offer excellent optical qualities and are, hence, applied in situations of highest aesthetic demands. Because of their lower mechanical stability, their indications are restricted to single crowns. Non-silica based ceramics, such as alumina and alumina-zirconia, exhibit higher mechanical stability.[6]

Yttria stabilized tetragonal zirconia polycrystals (Y-TZP) opens new vistas for all ceramic restorations. Yttria stabilized tetragonal zirconia polycrystal (Y-TZP) is a polymorphic material with three different allotropes- monoclinic, tetragonal and cubic.

Inserting force on its surface can lead to transition between its different crystalline reticulations that produce a volumetric change and create compressive stresses that seal the cracks. Due to the transformation toughening mechanism, Yttria stabilized tetragonal zirconia polycrystal (Y-TZP) has been shown to have superior mechanical properties compared to other all-ceramic systems[7].

Yttria stabilized tetragonal zirconia polycrystal (Y TZP) is currently used as a core material in full-ceramics dental restorations, implant superstructures and orthodontic brackets. High flexural strength and fracture toughness afford its application as framework material for fixed partial dentures even in loaded reconstructions in molar regions.[8]

With the introduction of modern technologies such as Computer Aided Design/Computer Aided Manufacture (CAD/CAM), fabrication of core designs for all-ceramic restorations have been revolutionized. Zirconia blocks can be milled at three different stages: green, pre-sintered, and fully sintered. The original frameworks milled from green-stage and pre-sintered zirconia blocks are enlarged to compensate for prospective material shrinkage that occurs during the final sintering stage.

The milling of green-stage and pre-sintered zirconia blocks is faster and causes less wear and tear on the milling tools than the milling of fully sintered blocks. Although fully sintered zirconia materials are extremely difficult and time-consuming to machine due to the increased hardness of the material, they are not subject to dimensional changes such as shrinkage after milling.[9,10]

According to various studies conducted failure rate of zirconia veneering ceramics were high especially when it comes to long term clinical success which is due to insufficient bond strength.[11] Clinical failures of veneered Yttria stabilized tetragonal zirconia polycrystalline (Y-TZP) frameworks due to chipping of the veneered ceramic were reported to be 13% on observation period over three years and 15.2% after five years.[11,12]

Sufficient bond strength between the veneering ceramic and the zirconia core is a concern for the long-term clinical success of zirconia restorations. Bond strength is determined by various host factors such as chemical bond strength, type of mechanical inter-locking and concentration of defects at the interface.

Other factors like wetting properties and degree of compression in the veneering layer due to difference in coefficients of thermal expansion between zirconia and the veneering ceramic also affect the bond strength.[13,14,15]

Even though various studies were carried out to find the influence of different surface treatments on bond quality, the researchers were not able to explain the mechanism of bonding between zirconia and veneering ceramic.

Sandblasting is a popular method to achieve the purpose of enhancing bond strength by increasing surface roughness and providing undercuts.[16,17,18] It also initiates phase transition, thus affecting mechanical strength and bonding capacity of the material.[19,20]

Laser pre-treatment has been a new approach for creating surface roughness without phase transformation in zirconia. The application of laser irradiation as a method of zirconia surface conditioning still lacks investigation.

Sufficient bond strength between the veneering ceramic and the substructure is a concern for the long-term clinical success of zirconia restorations. Hence the bond strength between the veneering ceramic and the zirconia is of utmost importance. Studies showed that sandblasting with air-borne particle abrasion and liner application increase the bond strength between the veneering ceramic and the zirconia.[21]

The purpose of this study is to evaluate and compare the shear bond strength of veneering ceramic to zirconia after surface treatment with sandblasting procedures and laser treatment.

AIMS AND OBJECTIVES

AIMS

- To determine and compare the shear bond strength of veneering ceramic to zirconia core without surface treatment and with various surface treatments.

OBJECTIVES

- To determine the shear bond strength between zirconia core and veneering ceramic without surface treatment.
- To determine the shear bond strength between zirconia core and veneering ceramic after air abrasion with 110-micron Alumina (Al_2O_3) particles on core surface.
- To determine the shear bond strength between zirconia core and veneering ceramic after Nd YAG laser treatment on core surface.

- To compare the shear bond strength of veneering ceramic to zirconia core

    i. without surface treatment,

    ii. with air abrasion and

    iii. with Nd YAG laser treatment.

REVIEW OF LITERATURE

Kosmac T et al (2000) [22] evaluated the strength and reliability of yttria stabilized zirconia after surface treatment with sandblasting and dental grinding and revealed, that dental grinding lowered the mean strength, whereas, sandblasting provided a powerful method for strengthening.

Isgro G et al (2003)[23] evaluated the effects of different surface treatments on the strength of a ceramic core material and veneering porcelain as well as influence of veneering porcelain on the strength of a two layer ceramic structure and concluded that the over glazed surface treatment significantly improved the strength of the core materials tested as well as the strength of two layer discs with veneer in tension.

Al-Dohan H M et al (2004) [24] evaluated the strength of the substructure and veneering porcelain interface in all ceramic systems and concluded that IPS-Empress2 with Eris, Procera All Zircon with CZR, DC Zircon with Vita were not significantly different from metal ceramic, the control group.

Guazzato M et al (2004)[25] evaluated the strength, fracture toughness and microstructure of DC Zirkon, In ceram zirconia slip and In ceram zirconia dry pressed were compared and concluded that zirconia based dental ceramics are stronger and tougher materials than conventional glass ceramics.

Aboushelib et al (2005)[26] evaluated the core-veneer bond strength and the cohesive strength of the components of three commercial layered all-ceramic systems and stated that polishing the core surfaces did not have an effect on the core-veneer bond strength and veneer with higher coefficient of thermal expansion (CTE) resulted in massive fractures in both the core and veneering material.

Guess P C et al (2008)[27] evaluated the shear bond strength between various commercial zirconia core and veneering ceramics after thermocycling and concluded that shear bond strength between zirconia core and veneering ceramics were not affected by thermocycling.

Texeria et al (2008)[28] studied basic fatigue parameters of a dental porcelain modified by deposition of yttria stabilized zirconia (YSZ) thin films and concluded that there was an increase in strength for specimens modified by application of yttria stabilized zirconia (YSZ) thin film.

Aboushelib et al (2008)[29] investigated the effect of zirconia type on the bond strength of two veneer ceramic and concluded that the addition of colouring pigments to zirconia frameworks resulted in structural changes that requires different surface treatment before veneering.

Fischer J et al (2008)[30] assessed the effect of different surface treatments on the bond strength veneering ceramics to zirconia and revealed that bonding between veneering ceramics and zirconia might be based on chemical bonds and also revealed that sandblasting was not necessary as a surface pre-treatment to enhance bond strength and that regeneration firing was not recommended.

Aboushelib M N et al (2008)[31] evaluated the reliable bond to zirconia based materials and revealed that combination of selective infiltration etching with experimental zirconia primers, significantly improved the micro tensile bond strength.

Sato H et al (2008)[32] evaluated mechanical properties of conventional yttria stabilized zirconia and ceria stabilized zirconia after sandblasting and heat treatment and concluded that ceria stabilized zirconia was more susceptible to stress induced transformation than yttria stabilized zirconia.

Choi B K et al (2009)[33] evaluated the bond strength of the porcelain veneer to the zirconia core and to other various metal alloys and concluded that there was significant difference in bond strength between the metal ceramic and zirconia but no significant difference in bond strength between the base metal and high noble metal alloys.

Nakamura T et al (2009)[34] examined the bond strength between tooth coloured porcelain and sandblasted zirconia framework at three different pressure (0.2 MPa, 0.4 MPa and 0.6 MPa) and concluded that zirconia specimens developed a strongest bond with tooth coloured porcelain when they were sandblasted at 0.4 MPa regardless of the type of porcelain.

Fischer J et al (2009)[35] assessed the effect of thermal misfit on the shear bond strength of zirconia/veneering ceramic composites and concluded that thermal expansion and glass transition temperature of the veneering ceramic have an impact on shear strength of veneer/zirconia composites.

Bulpakdi P et al (2009)[36] evaluated the failure analysis of clinically failed all-ceramic fixed partial dentures using fractal geometry and concluded that reason for early failures in fixed partial dentures could be occlusal overloading, stress corrosion, fatigue or improper structure design.

Karakoca S et al (2009)[37] evaluated the influence of surface grinding and sandblasting on surface roughness, phase transformation, and biaxial flexural strength of Y-TZP ceramics and concluded that phase transformation occurred in all groups after surface treatment, whereas, biaxial flexural strength decreased after grinding and increased after sandblasting.

Ahmed ATTIA et al (2010)[38] investigated the durability of repaired all ceramic crowns after cyclic loading and concluded that chair side treatment of fracture site by silica coating and silane application improved longevity of repaired In-Ceram zirconia crowns.

Nadia Z Fahmy et al (2010)[39] evaluated three veneering materials (Vitadur N, Vitadur Alpha, VM 7) for an all ceramic alumina system in terms of bond strength, micro hardness, and core/veneer interface quality and concluded that VM 7 showed highest shear bond value and lowest micro hardness value and also the perfect adhesion was between core and VM 7 veneering material.

Kawai Y et al (2010)[40] assessed the microstructure elemental distribution and crystal phase around the interface between zirconia and veneering porcelain using SEM-EDS (energy dispersed spectrometry) and micro-XRD (X-ray diffraction) and concluded that there was no phase transformation in the zirconia and porcelain even with extension firing period up to 384 hours suggestive of increased zirconia-porcelain bond.

Deng B et al (2010)[41] investigated the effects of different veneering porcelain on flexural strength of Yttria stabilized zirconia (Y-TZP) and summarized that for covering KaVo™ zirconia core material, conventionally applied veneering slurry-porcelain combined with liner or wash firing had significant higher bond strength than pressed-on porcelain and Scanning Electron Microscopy (SEM) showed that main failure type at the interface was adhesive.

Bonfante E A et al (2010)[42] checked the reliability and fracture patterns of zirconia veneered with pressable ceramic submitted to either axial or off axial contact fatigue and concluded that reliability was not significantly different between axial and off axis mouth motion fatigued pressable porcelain over Yttria stabilized tetragonal zirconia (Y TZP) cores.

Doi M et al (2011)[43] assessed the influence of pre-treatment of zirconia by sandblasting and or heat treatment on the flexural strength of zirconia and de-bonding or crack-initiation strength of porcelain-veneered zirconia ceramic and they concluded that the pre-treatment of zirconia with heat treatment after sandblasting prior to firing porcelain did not affect the de-bonding or crack initiation strength of porcelain- veneered zirconia ceramic.

Kim H J et al (2011)[44] examined the effects of various surface treatment on the shear bond strength of zirconia and veneering ceramic and concluded that shear bond strength of veneering ceramic on zirconia treated with airborne particle abrasion was significantly higher than that subjected to liner applied treatments.

Baldassarri M et al (2012)[45] evaluated the type and magnitude of residual stress on porcelain veneers of full contour fixed dental prostheses with an anatomic zirconia coping design and revealed the presence of radial tensile stress in the overlay porcelain, which in turn contributed to the large clinical chip fracture observed in prostheses.

Rismanchian M et al (2012)[46] evaluated the shear bond strength of two commercial zirconia core ceramics to their corresponding veneering ceramics and concluded that shear bond strength of Biodenta and Cercon were nearly same but the fracture mode of these two systems were different, so that the Biodenta porcelain was stronger than Cercon porcelain.

Sun Ting et al (2012)[47] investigated on the bond strength between various commercial ceramic core materials and veneering ceramics of dental bi-layered ceramic combinations (white cercon, yellow cercon, white lava, yellow lava, IPS e-max) and the effect of thermo-cycling and concluded that lithium di silicate based combinations produced the highest core- veneer bonds that overwhelmed the metal ceramic combinations and also the thermocycling and adding of pigments had no effect on core veneer bond strength.

Durand J C et al (2012)[48] evaluated the influence of a liner and regeneration firing at the interface between the zirconia core and veneering ceramic using confocal Raman microscopy and Energy Dispersive X- ray spectroscopy (EDS) and concluded that no substantial differences appeared in their chemical elemental composition, but the additional firing of the core decreased the inter-diffusion zone and the highest firing temperature of the liner increased the inter-diffusion zone.

Teng J et al (2012)[49] evaluated whether conditioning method improve core-veneer bond strength of zirconia restorations by measuring the shear bond strength and concluded that modifying the zirconia surface with powder coating could significantly increase the shear bond strength of zirconia to veneering porcelain.

Tan Sui et al (2013)[50] investigated the mechanisms of failure in porcelain-veneered zirconia restorations using Environmental Scanning Electron microscopy (ESEM) with Energy Dispersive X-ray (EDX) analysis and summarized that the failure was primarily due to fracture of veneering layers, which indicated the dominance of cohesive failure mode.

Saka M et al (2013)[51] evaluated the bond strength of veneer ceramic and zirconia cores with different surface modifications after microwave sintering and concluded that there was no difference in shear bond strength between conventionally sintered and microwave sintered specimen and adhesive failure was not seen in any specimens.

Ning xu et al (2013)[52] determined the effect of prolonged holding time in firing schedules on the bond strength between the zirconia core and veneered porcelain and concluded that prolonged holding time in firing schedule will improve the bond strength of zirconia core and veneered porcelain which reduce chipping failure of zirconia based all ceramic restorations.

Zeighami S et al (2013)[53] evaluated the effect of multiple firings on microtensile bond strength of core-veneer zirconia based all ceramic restorations and concluded that increase in firing cycles decreased the microtensile bond strength and most of the failures were cohesive in the veneering porcelain and did not change as the number of firing cycles increased.

Turp V et al (2013)[54] evaluated the effect of sandblasting with different particle sizes on the surface roughness and phase transformation of Yttria-stabilized tetragonal zirconia ceramics (Y-TZP) and summarized that 250 µm

Al_2O_3 particles for 30 seconds had the highest surface roughness and a significantly higher amount of monoclinic phase, compared to air abrasion with 30 µm ,50 µm and 110 µm Al_2O_3 particles.

Eroglu Z et al (2013)[55] evaluated the fatigue behavior of zirconia-ceramic, galvano-ceramic, and porcelain fused to metal fixed partial dentures before and after artificial fatigue testing and concluded that zirconia ceramic specimen was not significantly affected by fatigue whereas galvano-ceramic and porcelain fused to metal were affected.

Liu et al (2013)[56] investigated the effect of surface treatments in enhancing porcelain zirconia bonding and concluded that both sandblasting and CO_2 laser treatment increased the bond strength between porcelain and zirconia. X-ray diffractometry (XDR) analysis showed that sandblasting causes tetragonal to monoclinic phase transformation and regeneration firing reversed this transformation, however crystallographic transformation could not be detected in CO_2 laser treated specimens.

Grigore A et al (2013)[57] assessed the microstructure of veneered zirconia using transmission electron microscopy (TED) after surface treatments like thermal etching, sandblasting and coarse bur drilling and concluded that a reverse transformation of already transformed monoclinic zirconia grains, back into tetragonal polymorph has been observed after thermal veneering treatment.

Chintapalli R K et al (2013)[58] investigated the phase transformation and subsurface damage in zirconia after sandblasting and summarized that sandblasting induced monoclinic volume fraction in the range of 12- 15 % on the surface upto a depth of 12 ± 1 µm and this effect is found to be larger in specimen, sandblasted with large particles.

Miyazaki T et al (2013)[59] reviewed the current status of zirconia restorations and stated that there was no evidence demonstrating the presence of chemical bonding between zirconia and veneering ceramics and the major role in zirconia to porcelain integration was assumed to be mechanical bonding.

Aboushelib M N et al (2013)[60] evaluated the influence of different surface treatments on crystal structure and fracture strength of zirconia veneered restorations and concluded that abrasion with 50 µm particle size increased the flexural strength whereas abrasion with 120 µm particle size reduced the flexural strength due to higher percentage of monoclinic phase, however annealing reduced initial failure load for both particle size due to complete reverse transformation of monoclinic phase.

Yoon H et al (2014)[61] investigated the effect of surface treatment and liner material on the adhesion between veneering ceramic and zirconia and summarized that combination of surface sandblasting and bonding with liner increased the bond strength at the zirconia-veneer interface and the increase in bond strength varies for different types of liner materials.

Wang G et al (2014)[62] evaluated the effect of zirconia surface treatment on zirconia-veneer interfacial toughness, evaluated by fracture mechanics method and concluded that the toughness of zirconia-veneer interface with no treatment is significantly higher than that of interfaces subjected to liner application and airborne particle abrasion.

Korkmaz F M et al (2014)[63] studied the effect of surface treatment on the bond strength of veneering ceramic to zirconia and concluded that bond strength of metal primer treated zirconia specimens were significantly higher than sandblasting, Clearfil ceramic primer, grinding and Relyx ceramic primer groups. SEM analysis demonstrated that metal primer treated zirconia specimens had mainly cohesive failures while other groups showed mainly equal level of adhesive and mixed fracture types.

Sun T et al (2014)[64] evaluated the shear bond strength comparison between conventional dental nano zirconia combinations and new functionally graded nano zirconia combinations after thermal mechanical cycling and summarized that functionally graded zirconia combinations exhibit greater shear bond strength than zirconia combinations irrespective of fatigue conditions.

Anami L C et al (2014)[65] evaluated the influence of the geometry and design of zirconia crown preparation on stress distribution, using finite element analysis and concluded that modified design of the zirconia copings reduces the stress contribution at the interface with the veneer-ceramic, and the simplified preparation can exert a stress distribution similar to that of anatomical preparation at and near the load point, when load is applied to the center of the crown.

Costa A K et al (2014)[66] evaluated the strength of all ceramic restorative systems introduced using CAD CAM technology to fabricate both zirconia core and veneer ceramic layers with that of conventionally sintered zirconia core ceramic veneer and concluded that CAD CAM produced zirconia core ceramic veneer appears to offer the potential to induce more favourable stress within veneer-ceramic when compared with conventional sintered manufacturing routes.

Schneider G A et al (2015)[67] evaluated the adhesion of porcelain to zirconia by Schwickerath adhesion test and concluded that the minima of fracture test provide a simple basis for the calculation of adhesion toughness and exhibits far lesser scatter than the load to initiate fracture.

Subash M et al (2015)[68] evaluated the shear bond strength between zirconia core and ceramic veneers fabricated by pressing and layering techniques and concluded that pressed ceramic performed better than layered specimen.

Wendler M et al (2015)[69] evaluated the coefficient of thermal expansion mismatch and the cooling protocol on the distribution of residual stress and crack propagation in veneered zirconia bilayers and concluded that higher coefficient of thermal expansion generated an important stress gradient with high compressive residual stresses near the interface, hindering the crack propagation.

Inokoshi M et al (2016)[70] analyzed the zirconia- veneering ceramic interface structurally and chemically using Field emission gun SEM (Feg-SEM), Scanning transmission electron microscopy (STEM), micro Raman spectroscopy and High resolution transmission electron microscopy (HRTEM) and concluded that even though there was minor elemental shift, there was no definitive conclusion regarding the chemical bond strength between veneering ceramic and zirconia.

Yoon H et al (2016)[71] analyzed- the effect of various surface treatments on the interfacial adhesion between zirconia cores and porcelain veneers and concluded that surface treatments with intermediate ceramics maybe beneficial to the interfacial adhesion between the zirconia cores and veneering porcelains and the crack failure mode was adhesive for all specimens.

Madani A et al (2016)[72] evaluated the effect of silica and aluminosilicate nanocomposite coating of zirconia based dental ceramic by sol gel dip coating technique on the bond strength of veneering porcelain to Yttria stabilized zirconia and concluded that micro-tensile bond strength of aluminosilicate sample were significantly high compared to control and sandblasted groups.

MATERIALS AND METHODOLOGY

In the present study an effort was made to find out the shear bond strength of veneering ceramic over zirconia after pre-treatment with sandblasting and laser.

MATERIALS:

1) Zirconia blocks. (Katana HT, Kuraray Noritake Dental, Japan from Confident Pvt. Ltd. Bangalore, India).

2) Instruments used for veneering ceramic over zirconia

   i) Kolinsky brush No.6 (Renfert, Germany)

   ii) Ceramic humidor (Yeti Dental, Germany)

3) Veneering Ceramic - VITA VM9 (Vita Zahnfabrik, Germany).

4) Alumina 110 micron, (Aluminox, Germany).

5) Demineralized water (Medilise Chemicals, India).

EQUIPMENTS:

1) Design Software - Magics Software.

2) Milling Machine - MB Maschinen, Germany.

3) Sintering furnace - Mihm Vogt, Germany.

4) Ultrasonic cleaner - Confident C-80-L, India.

5) Sandblasting machine - Delta Dual Blaster, India.

6) Nd YAG Laser - Fotona, Solvenia.

7) Sintering furnace - Ivoclar vivadent programat p300, Germany.

8) Universal testing machine- Instron 3345, USA.

METHODOLOGY

Methodology of this study is been divided into following stages:

1. Sample selection.
2. Sample size.
3. Sample preparation.
4. Sample grouping.
5. Measurement.

SAMPLE SELECTION:

Zirconia block from Katana HT, Kuraray Noritake Dental, Japan and ceramic from Vita VM 9 (Vita Zahnfabrik, Germany) were selected for this study.

SAMPLE SIZE:

$$n = \frac{Z^2 S^2}{d^2}$$

Z- value associated with confidence = 1.96

S- Standard deviation of sandblasting treated group = 36.63

d- Absolute precision = 2.96

Sample size (n) = 23.37

= 24

In this study sample size is 30.

SAMPLE PREPARATION:

Thirty zirconia samples (Katana HT, Kuraray Noritake Dental, Japan from Confident Pvt. Ltd. Bangalore, India) were fabricated using CAD technique and divided into three groups. After surface treatment of the sample ceramic Vita VM 9 were veneered onto the sample by conventional layering method.

SAMPLE GROUPING:

Group I: Control group (10 samples)

Group II: Sandblasted with 110µ alumina group (10 samples)

Group III: Nd YAG laser treated group (10 samples)

PREPARATION OF ZIRCONIA SAMPLES:

The needed dimension of zirconia block was designed using Design software (Magics Software). The file created using Magics software was saved in STL format. As per the data present in the STL file commercially available 3Y-TZP block (Katana HT, Kuraray Noritake Dental, Japan from Confident Pvt. Ltd. Bangalore, India) was machined to 7 mm thickness, 7 mm breadth and 15 mm length rectangular samples using milling machine (MB Maschinen, Germany). These samples were sintered using sintering furnace (Mihm Vogt, Germany). Then, these samples were cleaned ultrasonically using Ultrasonic cleaner (Confident C-80-L) and further cleaned with demineralized water, followed by drying in hot air.

SURFACE TREATMENT OF ZIRCONIA SMAPLES

After milling and sintering of the zirconia they are cleaned using ultrasonic cleaner (Confident C-80-L) followed by drying in hot air. These samples are then surface treated specifically into the areas of veneering ceramics and was divided accordingly:

i) Sandblasting with 110-micron alumina particles.

ii) Laser treatment with Nd YAG laser.

Sandblasting with 110 μ alumina particle size:

Zirconia samples were sandblasted in Delta Dual Master with air abrasion from 110-micron alumina particles at 2 bar pressure at a distance of 10 mm for 5 seconds.

Laser treatment with Nd YAG laser:

Zirconia samples were surface treated with Nd Yag laser (Fotona Fidelis Plus III Solvenia) using an optical fiber of 300 μm diameter in contact mode. The laser settings were 2 Watt (W) peak power, 20 Hz frequency for a time period of 60 seconds creating a maximum energy of 120 Joules(J). The zirconia samples were surface treated twice using the specified laser settings.

Veneering ceramic to zirconia blocks

After surface treatment ceramic were veneered onto the zirconia block. Prior to adding ceramic to zirconia the samples were cleaned using ultrasonic cleaner (Confident C-80-L) and were air dried.

Veneering ceramic was done using a metal mould of 4 mm length 4 mm width and 4 mm thickness for dimensional accuracy and the technique followed was conventional layering method. The veneering ceramic powder was mixed with build-up liquid and the mix obtained was applied to the core using Kolinsky Brush No.6 brush followed by blotting with tissue paper to remove excess of liquid. Veneering was done in increments to improve accuracy that may occur due to shrinkage during sintering.

Firing chart for Vita VM9

VITA VM9	Low temp(ºC)	Dry time (sec)	Heat rate (ºC/min)	High temp (ºC)	Preheat (min)	Vaccum (min)
Effect bonder	500	6.00	80	980	1.00	6.00
Effect bonder paste	500	6.00	80	980	2.00	6.00
Base dentine	500	2.00	60	950	1.00	7.27
Effect liner	500	6.00	55	930	1.00	7.49
1st dentine	500	6.00	55	910	1.00	7.27
2nd dentine	500	6.00	55	900	1.00	7.16
Glaze	500	4.00	80	900	1.00	-
Corrective	500	4.00	60	760	1.00	4.20

MEASUREMENT:

Shear bond strength of veneering ceramic to zirconia was tested using Universal testing machine-Instron 3345, USA and the value obtained was recorded using Blue Hill 3 software. Scanning Electron Microscopy of the samples were done at 500X, 1000X, 2000X to evaluate the surface roughness on the zirconia surface.

FIGURES

Fig 1: ZIRCONIA MILLING MACHINE

Fig 2: THIRTY ZIRCONIA SAMPLES

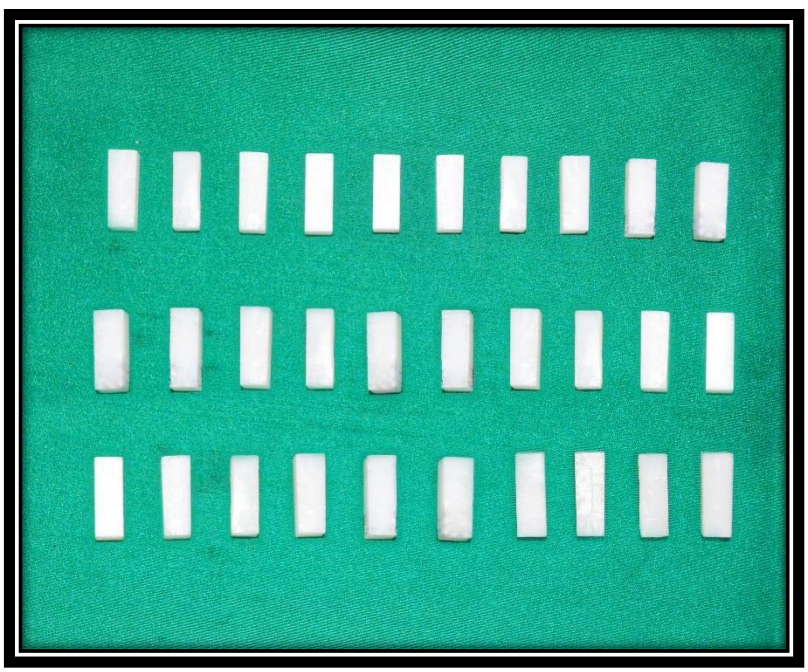


Fig 3: SAND BLASTER

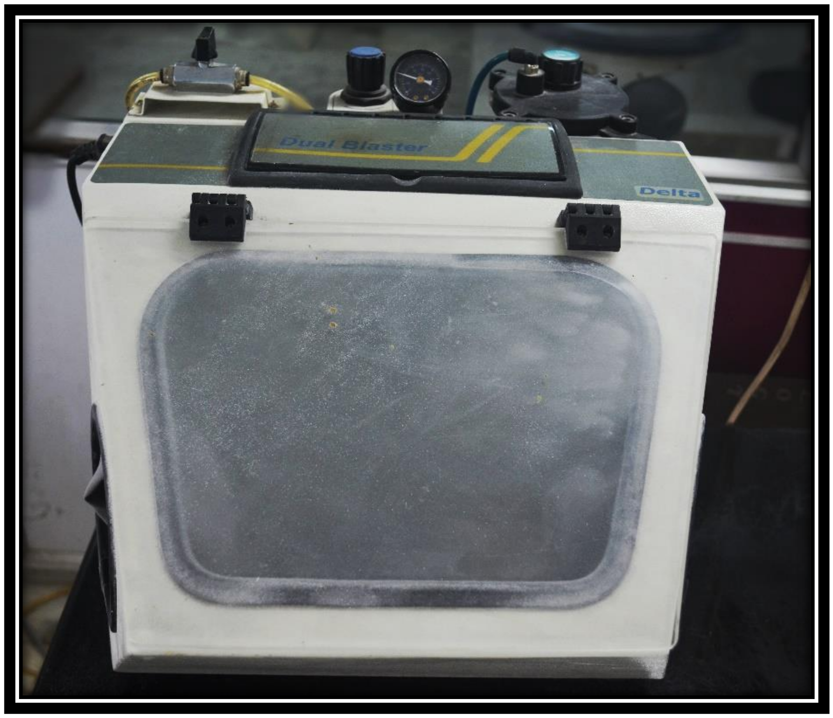


Fig 4: ALUMINOX

Fig 5: SAND BLASTING

Fig 6: FOTONA LASER

Fig 7: LASER VALUE SETTING

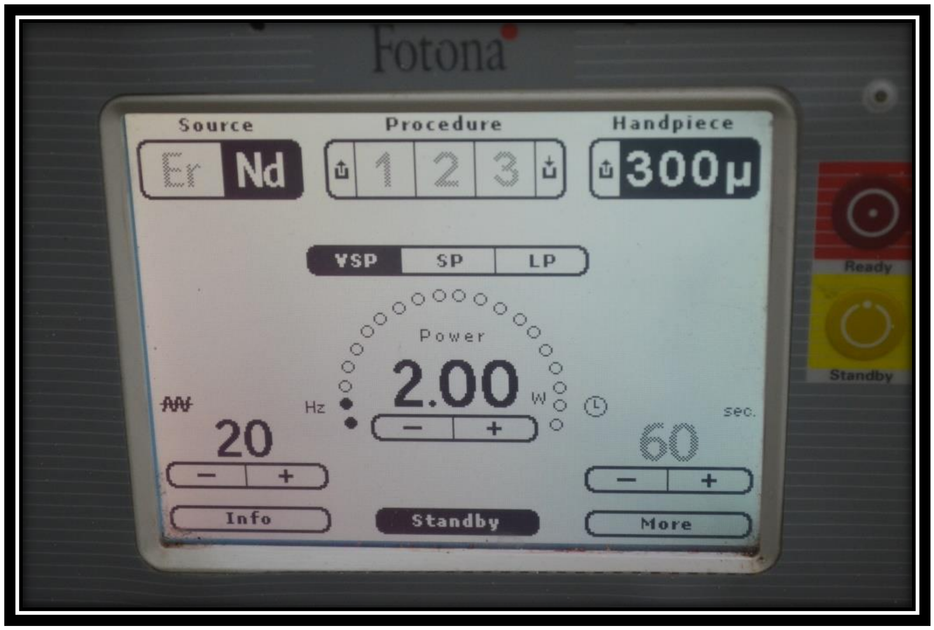


Fig 8: LASER ETCHING

Fig 9: ULTRASONIC CLEANER

Fig 10: DEMINERALIZED WATER

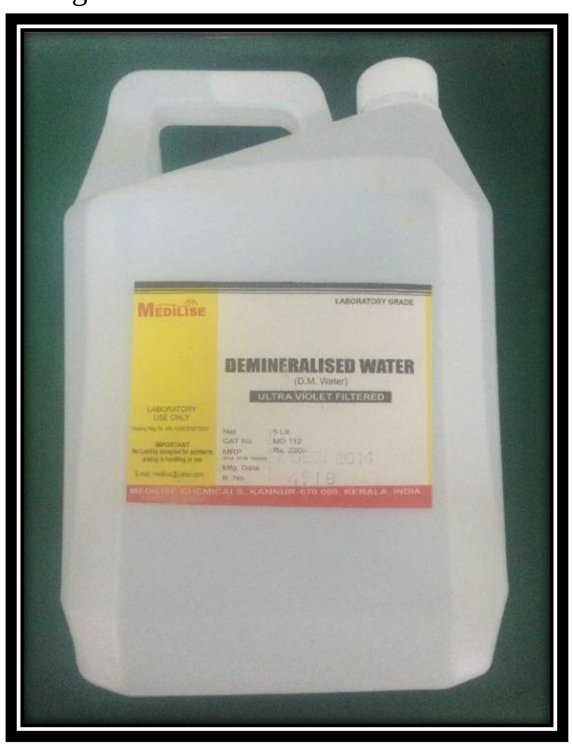


Fig 11: ULTRASONIC CLEANING

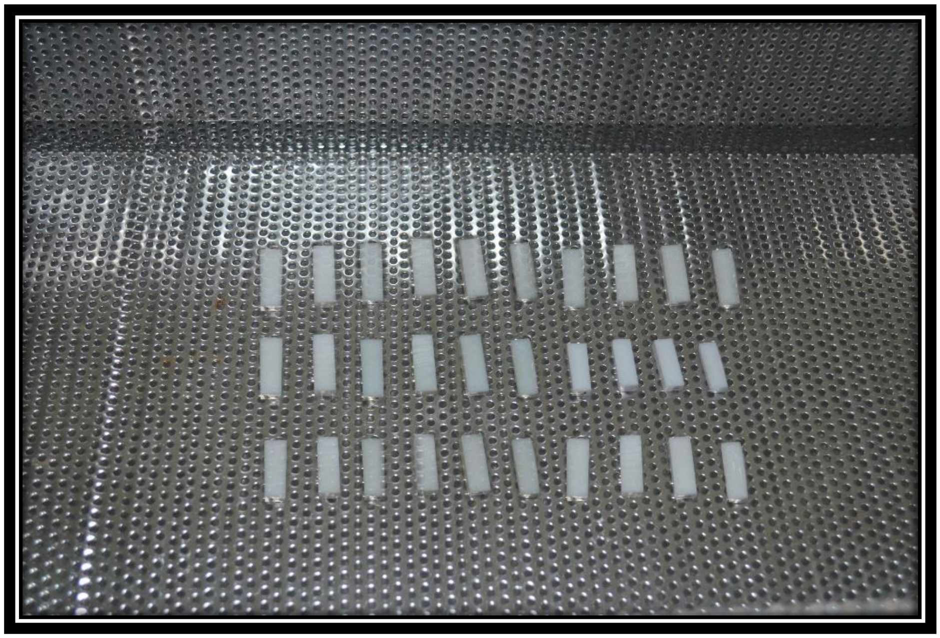


Fig12: VITA CLASSIC VM9

Fig 13: UNIVERSAL BUILDING LIQUID

Fig 14: KOLINSKY BRUSH

Fig15: CERAMIC HUMIDOR

Fig16: CERAMIC LAYERING

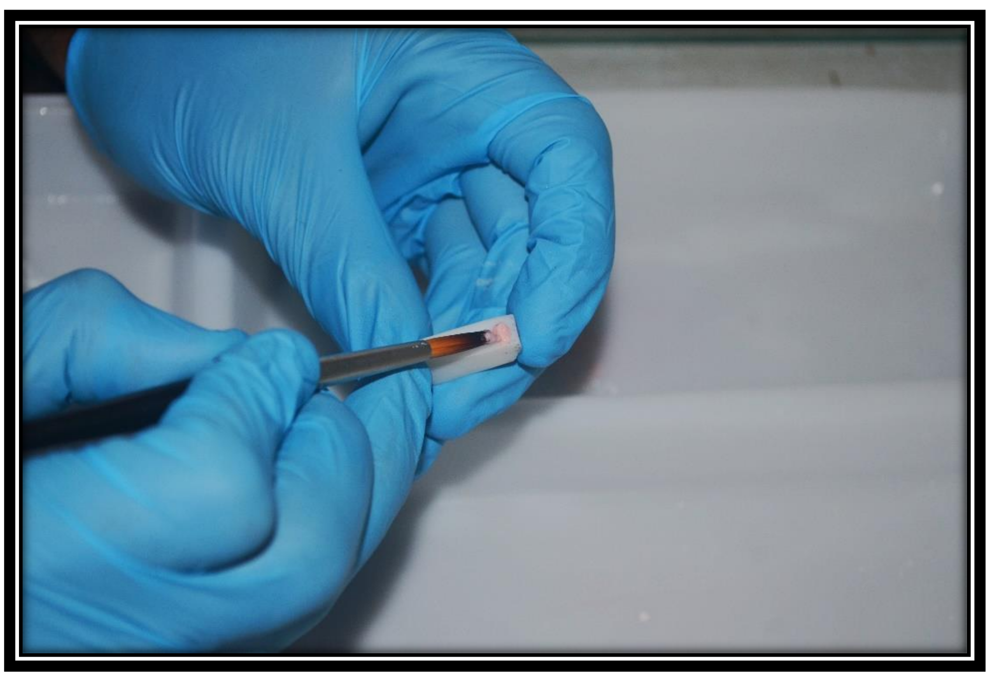


Fig17: SINTERING FURNACE

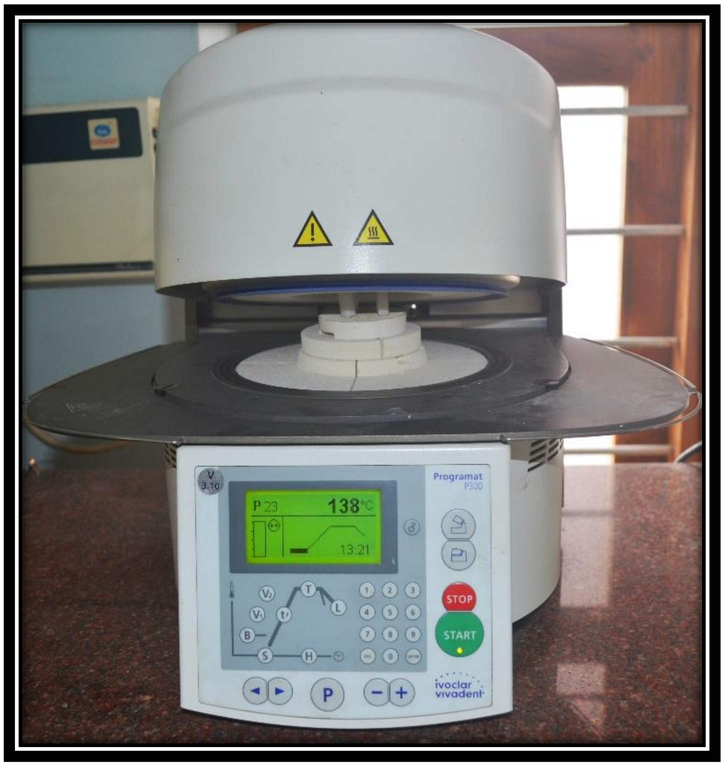


Fig 18: CERAMIC VENEERED ZIRCONIA

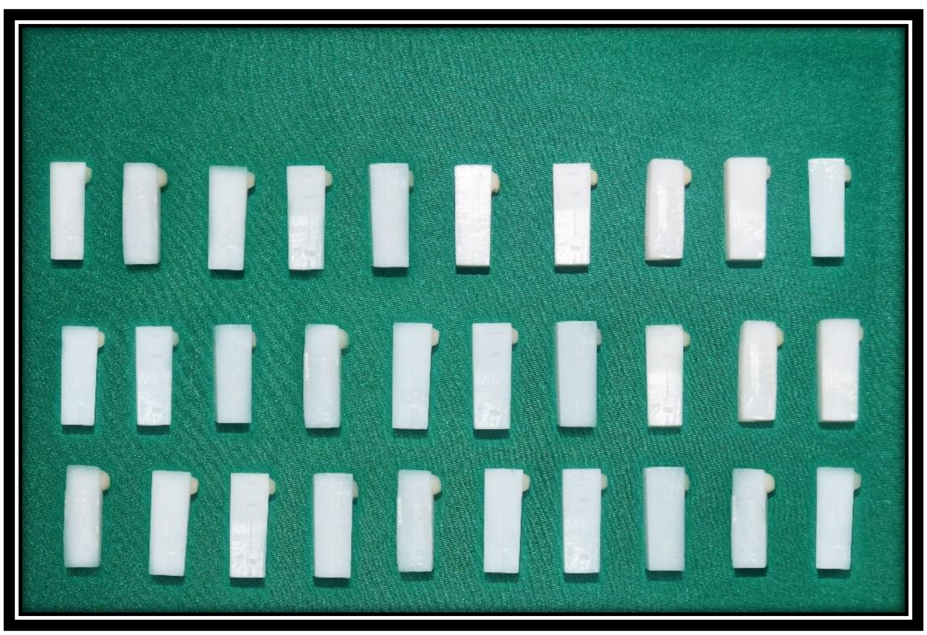


Fig19: AIROTOR

Fig 20: UNIVERSAL TESTING MACHINE

Fig 21: SAMPLE LOADED ON UNIVERSAL TESTING MACHINE

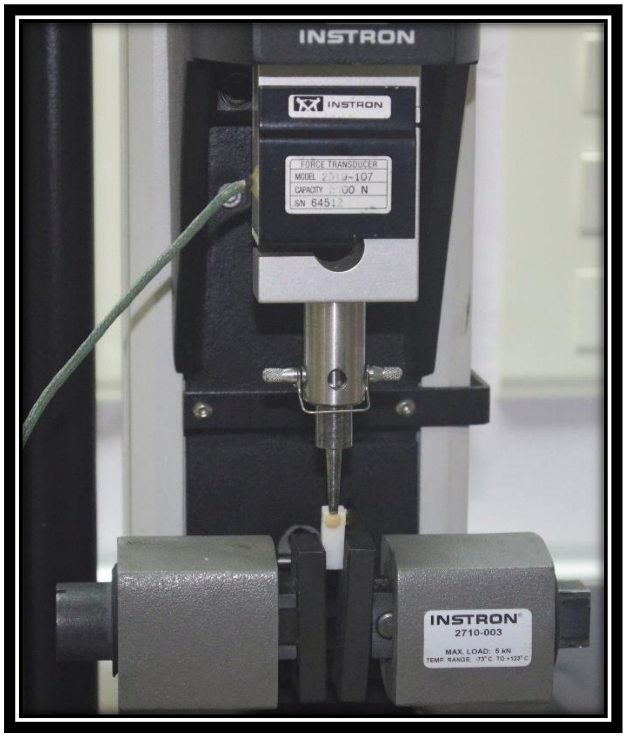


Fig22:ION SPUTTER

Fig 23: GOLD SPUTTED SAMPLES

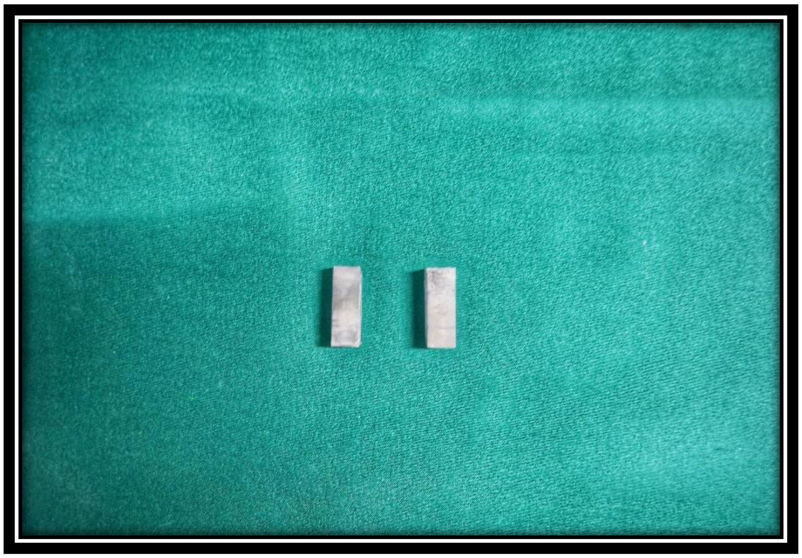


Fig 24: SCANNING ELECTRON MICROSCOPE

RESULTS

STATISTICAL ANALYSIS

The data was expressed in mean and standard deviation (MEAN±SD). Statistical Package for Social Sciences (SPSS 16.0) version used for analysis. ANOVA (Post hoc) followed by Dunnett 't' test applied to find the statistical significant between the groups. P value less than 0.05 ($p<0.05$) considered statically significant at 95% confidence interval.

Table – 1 shows the mean values of different groups.

Table – 2 shows the comparison of Mean value of group I (Non treated zirconia blocks – control) with group II (Sandblasted zirconia blocks) and III (Nd YAG laser treated zirconia blocks). Group II and III shows significant values ($P < 0.05$) when compared with group I.

Table – 3 shows the comparison of mean value of group II with other groups III and I. Group III and I shows significant value ($P < 0.05$) when compared with group II.

Table – 4 shows the comparison of mean value of group III with groups I and II. Group I and II shows significant value (P < 0.05) when compared with group III.

Table – 5 shows multiple comparison of mean value between Zirconia groups. Group I shows significant values (P < 0.05) when compared with other groups I and II, group II shows significant values (P < 0.05) when compared with other groups III and I, and group III shows significant values (P < 0.05) when compared with other groups I and II.

TABLES

Table-1: Mean Load and Shear Bond strength values of different groups

Groups	Treatment	Load(N) (MEAN±SD)	Shear Bond Strength (MPa) (MEAN±SD)
Group-I	**Control**	454.51±4.49	28.30±2.92
Group-II	**Sand blast**	545.52±3.99	33.92±2.67
Group-III	**Laser treatment**	666.90±8.27	41.67±5.16

Table-2: Comparison of mean load and shear bond strength value of Group-I with other groups

Groups	Max Load (MEAN±SD)	P value	Shear Bond Strength (MPa) (MEAN±SD)	P value
Group-I	454.51±4.49		28.30±2.92	
Group-II	545.52±3.99*	**0.001**	33.92±2.67*	**0.03**
Group-III	666.90±8.27*	**0.001**	41.67±5.16*	**0.01**

(*p<0.05 significant compared Group-I with other groups)

Table-3: Comparison of mean load and shear bond strength value of Group-II with other groups

Groups	Load (MEAN±SD)	P value	Shear Bond Strength (MPa) (MEAN±SD)	P value
Group-II	545.52±3.99		33.92±2.67	
Group-I	454.51±4.49*	**0.001**	28.30±2.92*	**0.03**
Group-III	666.90±8.27*	**0.001**	41.67±5.16*	**0.01**

(*p<0.05 significant compared Group-II with other groups)

Table-4: Comparison of mean load and shear bond strength value of Group-III with other groups

Groups	Load (MEAN±SD)	P value	Shear Bond Strength (MPa) (MEAN±SD)	P value
Group-III	666.90±8.27		41.67±5.16	
Group-I	454.51±4.49*	**0.001**	28.30±2.92*	**0.01**
Group-II	545.52±3.99*	**0.001**	33.92±2.67*	**0.01**

(*p<0.05 significant compared Group-III with other groups)

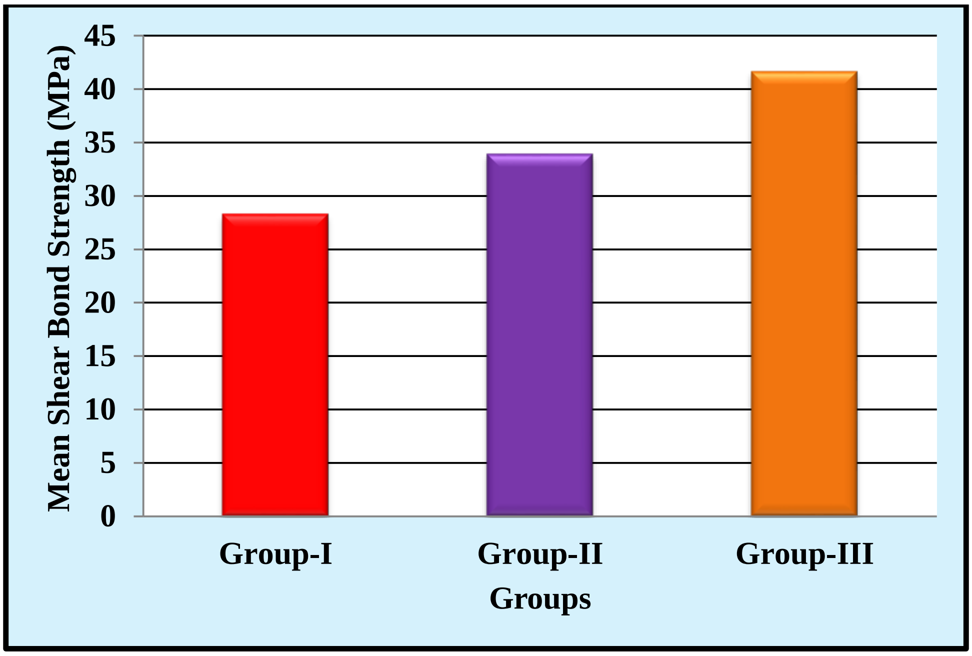
Table-5: Multiple comparison of mean load and shear bond strength value of different groups

Groups	Treatment	Load (MEAN±SD)	Shear Bond Strength (MPa) (MEAN±SD)
Group-I	**Control**	454.51±4.49	28.30±2.92
Group-II	**Sand blast**	545.52±3.99*	33.92±2.67*
Group-III	**Laser treatment**	666.90±8.27*,#	41.67±5.16*,#

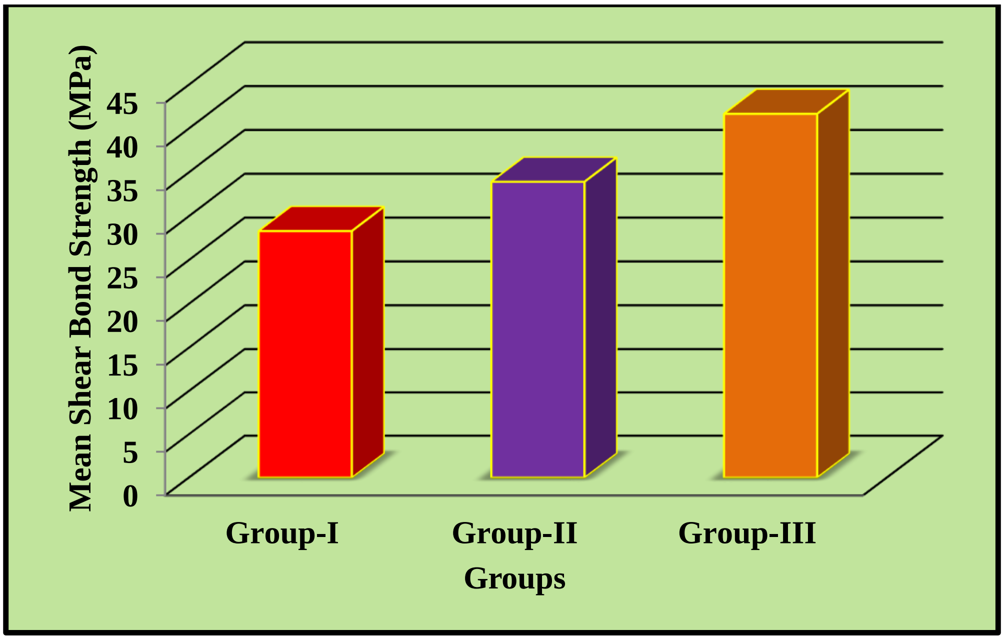
(*p<0.05 significant compared Group-I with other groups, #p<0.05 significant compared Group-II with other groups)

GRAPHS

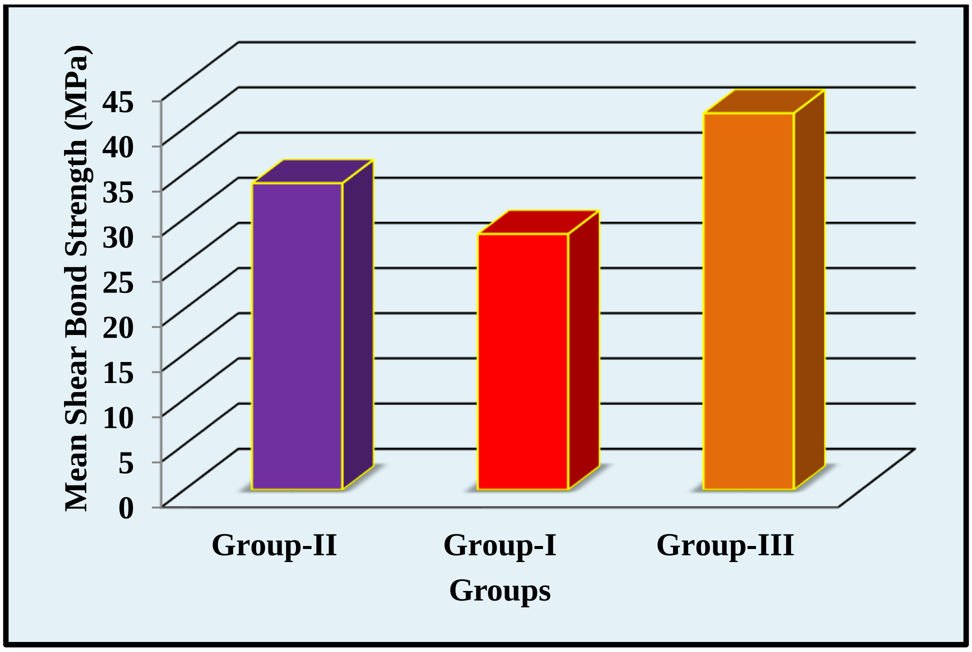
Graph-1: Mean Shear Bond Strength values of different groups



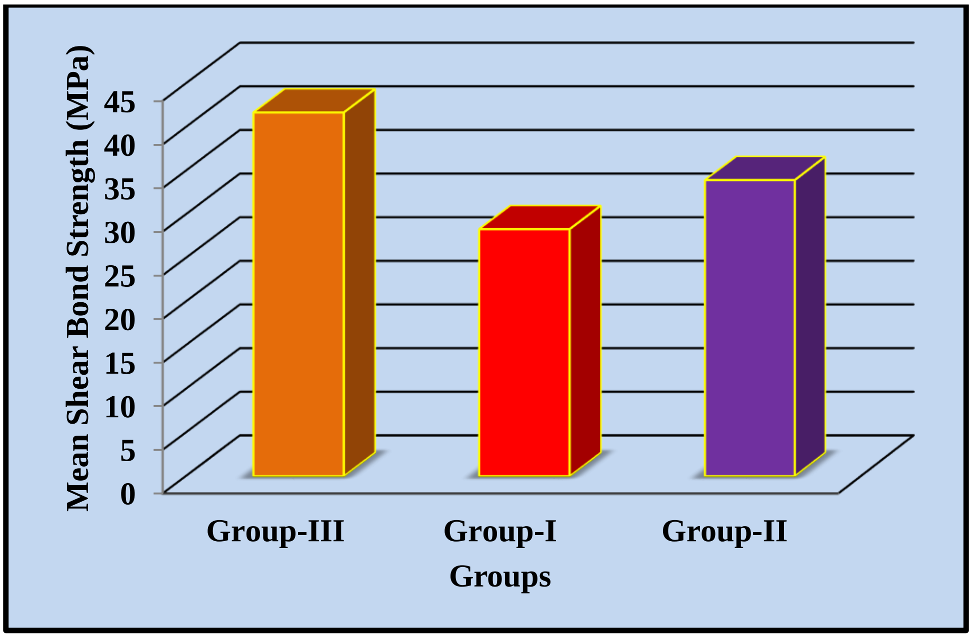
Graph-2: Comparison of mean load and shear bond strength value of Group-I with other groups



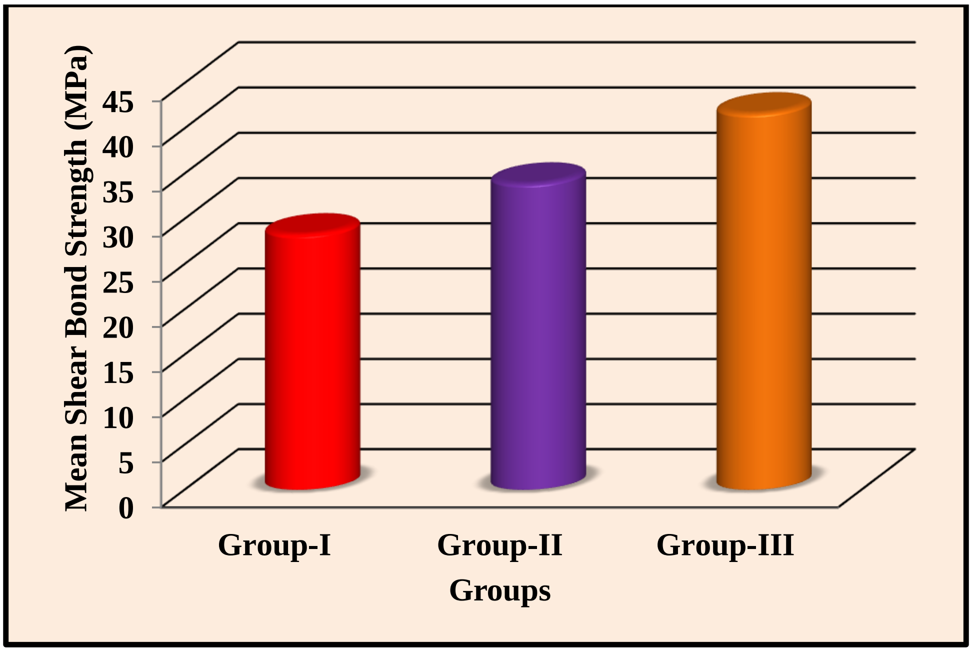
Graph-3: Comparison of mean load and shear bond strength value of Group-II with other groups



Graph-4: Comparison of mean load and shear bond strength value of Group-III with other groups



Graph-5: Multiple comparison of mean load and shear bond strength value of different groups



SEM RESULTS

SCANNING ELECTRON MICROSCOPIC ANALYSIS TO DETERMINE SURFACE ROUGHNESS

- Figure 25 represents the scanning electron microscopic image of group II under the magnification of 500X showing rough surface with irregular pits.

- Figure 26 represents the scanning electron microscopic image of group II under the magnification of 1000X showing rough surface with irregular pits.

- Figure 27 represents the scanning electron microscopic image of group II under the magnification of 2000X showing perceptible loss of Y-TZP particles.

- Figure 28 represents the scanning electron microscopic image of group III under the magnification of 500X showing uniform blister like appearance.

- Figure 29 represents the scanning electron microscopic image of group III under the magnification of 1000X showing flake like areas and melted areas.

- Figure 30 represents the scanning electron microscopic image of group III under the magnification of 2000X showing regular shallow pits with minimal loss of Y-TZP particles.

SEM FIGURES

FIG 25: SCANNING ELECTRON MICROSCOPE IMAGE OF SANDBLASTED ZIRCONIA AT 500X SHOWING ROUGH SURFACE WITH IRREGULAR PITS

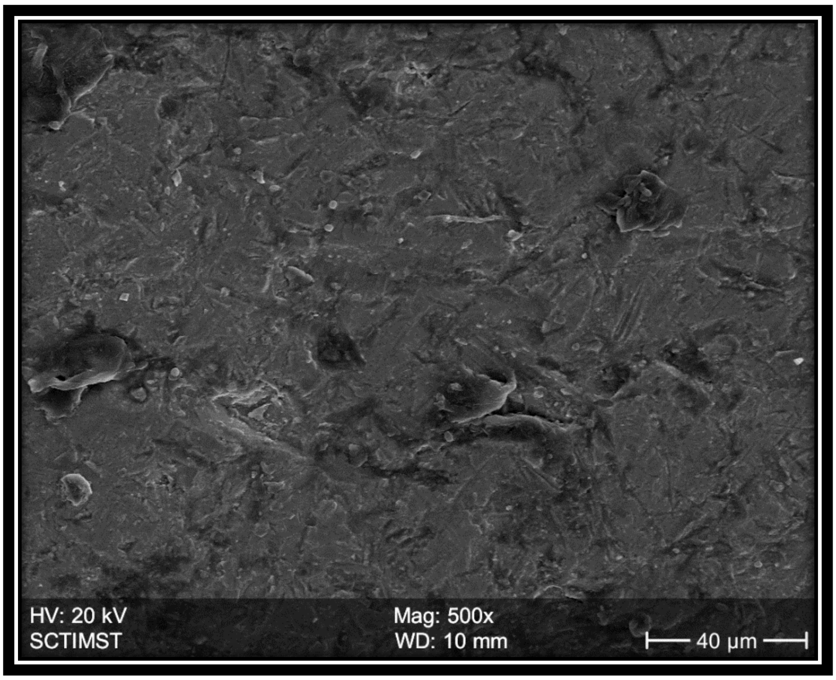


Fig 26: SCANNING ELECTRON MICROSCOPE IMAGE OF SANDBLASTED ZIRCONIA AT 1000X SHOWING ROUGH SURFACE WITH IRREGULAR PITS

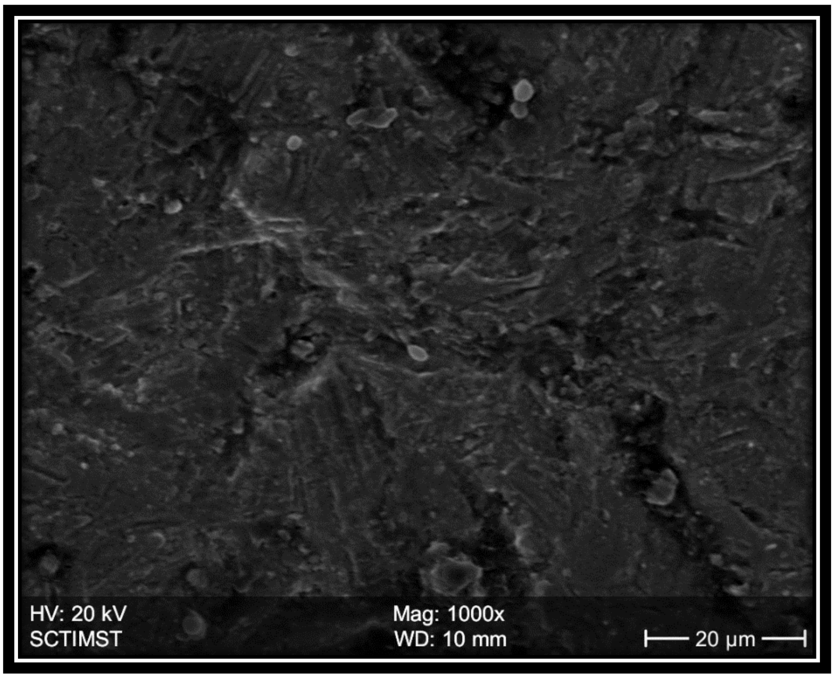


Fig 27: SCANNING ELECTRON MICROSCOPE IMAGE OF SANDBLASTED ZIRCONIA AT 2000X SHOWING PERCEPTIBLE LOSS OF Y-TZP PARTICLES

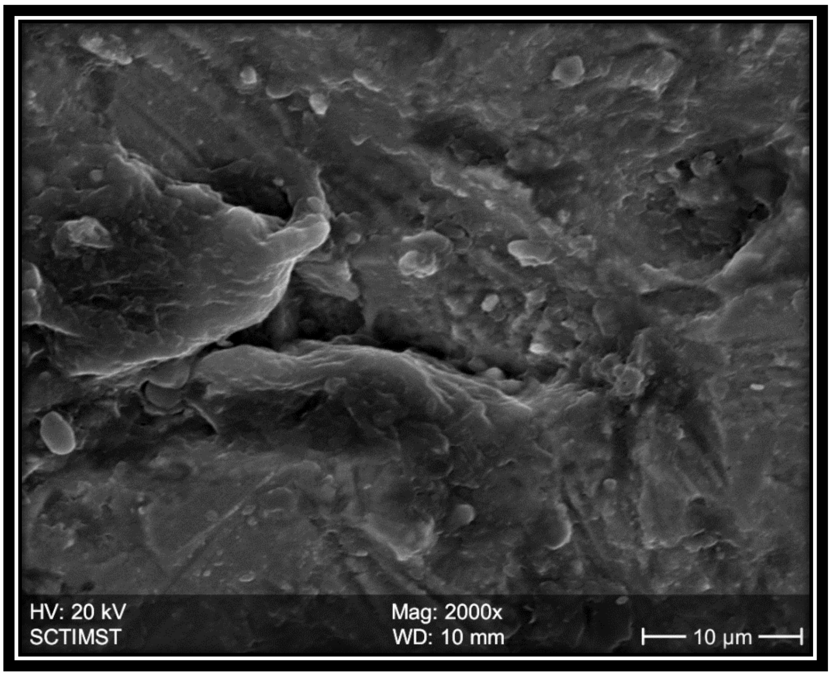


Fig 28: SCANNING ELECTRON MICROSCOPE IMAGE OF LASER TREATED ZIRCONIA AT 500X SHOWING UNIFORM BLISTER LIKE APPEARENCE

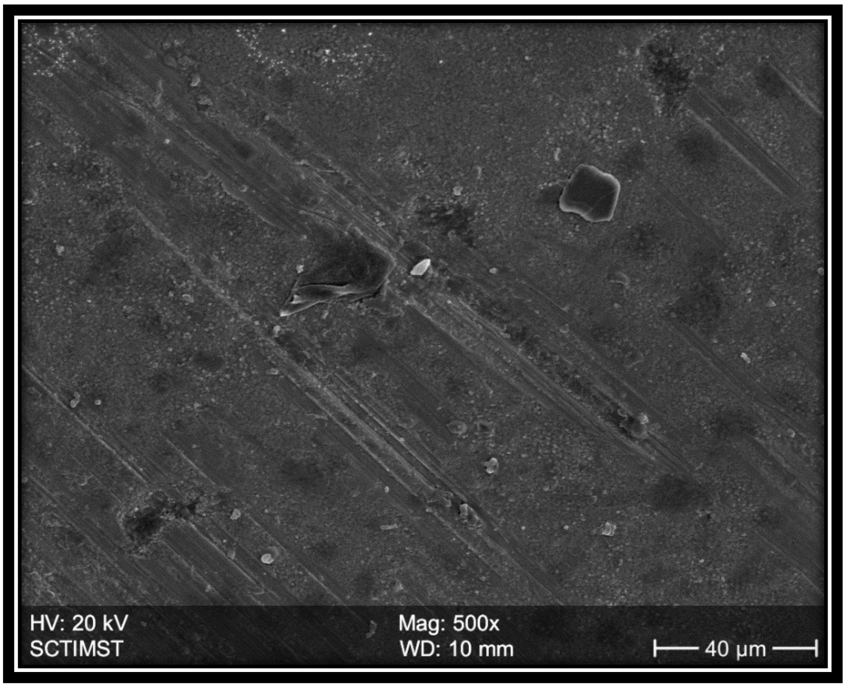


Fig 29: SCANNING ELECTRON MICROSCOPE IMAGE OF LASER TREATED ZIRCONIA AT 1000X SHOWING FLAKE LIKE AREAS AND MELTED AREAS

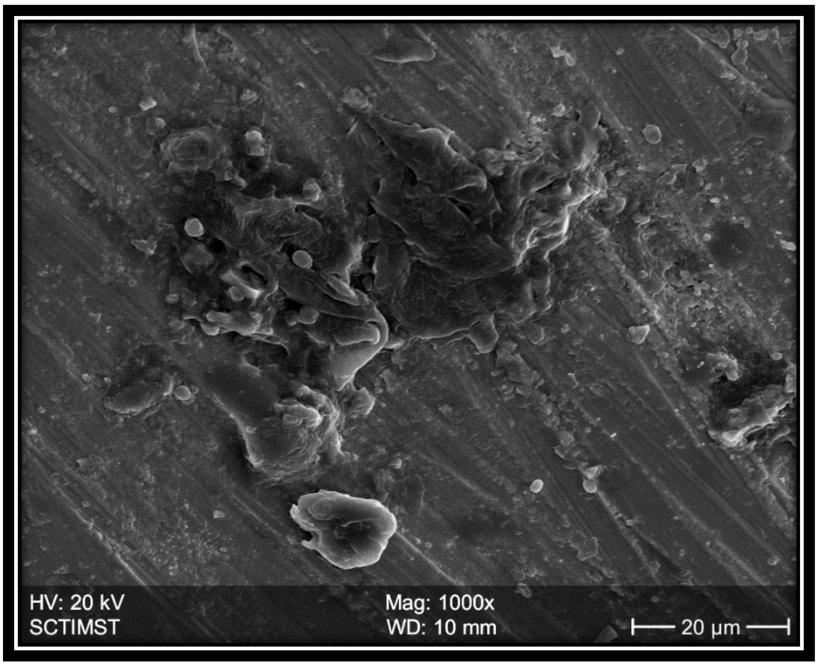
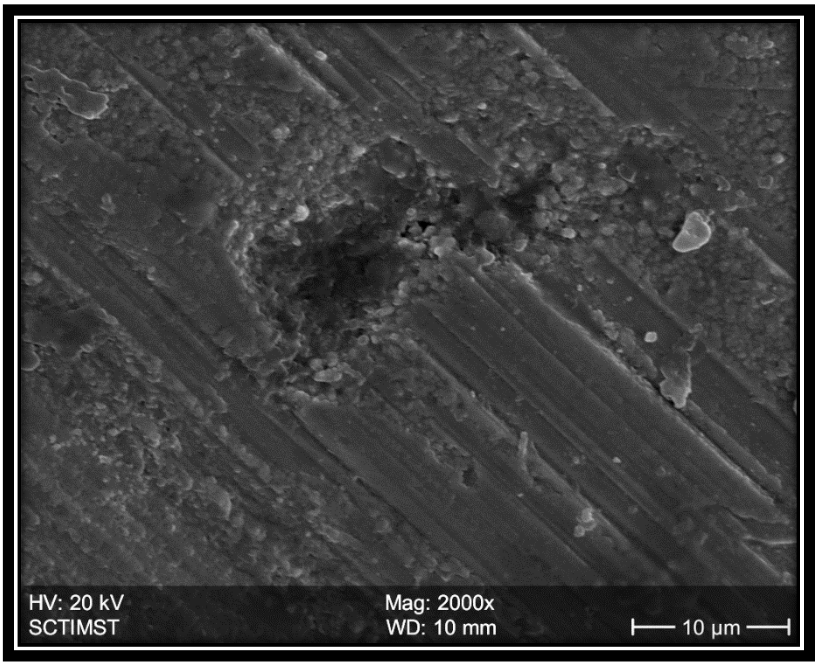


Fig 30: SCANNING ELECTRON MICROSCOPE IMAGE OF LASER TREATED ZIRCONIA AT 2000X SHOWING REGULAR SHALLOW PIT WITH MINIMAL LOSS OF Y-TZP PARTICLES



DISCUSSION

The present study demonstrated that the shear bond strength of surface treated groups were higher than the control group and also there was significant difference in bond strength between surface treated groups therefore null hypothesis was rejected. The results also suggested that laser treatment is more useful than sandblasting for increasing the bond strength between zirconia and veneering ceramic.

The shear bond strength was used frequently in many studies and reported to be relatively simple and easily performed.[73] Shear bond strength has not been standardized. There were various factors that affect shear bond strength test such as geometrical shape of the specimens, type of the substrates, storage conditions and cross-head speed.[26,74]

The International Organisation for Standardization (ISO) standards recommended that the rate of loading for a bonded specimen should be 0.75±0.30 mm/min.[75] In several studies for evaluating the shear bond strength the cross-head speed was set at 1mm/min.[35,76] In the present study the cross-head speed was set at 1mm/min.

The aim of surface treatment on the zirconia surface is to increase surface roughness at the microscopic level which ultimately results in high surface energy and thereby better wettability. [77,78]

Sandblasting is one of the frequently used method of surface treatment. Some manufacturers recommend sandblasting as a routine method of pre-treatment to increase the bond strength. In this study also increasing veneering ceramic to zirconia shear bond strength was concluded. However, in another study done by Fischer J et al sandblasting resulted in increased surface roughness of zirconia such treatment did not result in stronger veneering ceramic to zirconia bonding. Fischer J et al also stated that zirconia and veneering ceramic might bond chemically.[30]

There were several studies that investigated the influence of sandblasting on the bonding reliability of zirconia. These studies shown that sandblasting increase surface roughness which ultimately lead to the formation of a compressive layer on zirconia surface and thus increased the flexural strength. According to Grigore et al and Inokoshi M et al sandblasting damaged the zirconia surface upto a depth of 1 to 1.5 µm. [57,70,]

Sandblasting the zirconia surface leads to phase transformation on the surface of zirconia from stable tetragonal (T) to unstable monoclinic (M) phase. Even though monoclinic (M) phase was limited to a depth 0.33 µm, the occurrence of such crystallographic phase transformation might impair the long term reliability of such restorations.[37,79] Although in the present study sandblasting improved the bond strength between zirconia and veneering ceramic; alternative methods that not only improve bond strength but also have less effect on the stability of zirconia core are needed.

Laser treatment has been used in dental practice for many years. The mechanism for tooth tissue ablation is that when water contents in the tooth tissue absorb laser energy they will be turned into steam instantly and the resultant volume expansion will lead to micro-explosion which removes the ambient tissues and makes surface rougher. In zirconia there is no water content exists. The main effects of laser irradiation on zirconia may be melting and re-hardening of surface due to ample absorption of laser energy. These changes in zirconia surface topography results in different surface roughness values.[80,81]

The value of zirconia roughness after being irradiated with pulse mode Erbium Doped Yttrium Aluminium Garnet (Er: YAG) laser beam were claimed to be significantly higher. Although high laser energy had distinct effects on zirconia, low energy laser beam which cause less destructive effects was found to be beneficial.[82]

In one of the studies it was declared that resin zirconia shear bond was enhanced by pulse mode Erbium Doped Yttrium Aluminium Garnet (Er: YAG) laser beam and continuous mode CO_2 laser.[83] The difference in type of laser beam have different effect on zirconia bonding properties due to difference in laser energy adsorption.[84]

It is believed that core and veneer materials fuse together and some elements from each material diffuse across the interface. This concept was explained by Smith et al[85] and Al-Dohan et al.[24] Either of these occurrences can cause a chemical alteration of the glass layer adjacent to the core, possibly by altering the physical properties, such as strength or coefficient of thermal expansion at the interface. However, the precise bonding mechanism between zirconia and veneering ceramic has not yet been identified.

The shear bond strength measured in the present study ranged from 28.3±2.92 to 41.67±5.16. In metal ceramic restorations a bond strength greater than 25 MPa between the layering porcelain and metal is believed to be adequate according to International Organisation for Standardization (ISO); however, no such estimate for adequate bond strength in all ceramic materials has been determined.[86]

According to Guess et al,[27] the method applied to metal ceramic systems cannot be used in bi-layered ceramic materials due to brittleness. According to Aboushelib et al,[26] the micro-tensile bond strength test is a reliable assessment technique and Guess et al[27] confirmed that the Schmitz-Schulmeyer test was reliable due to minimal experimental variables. Although testing can be performed with a crown-shaped framework to reproduce the clinical situation, standardizing the various testing designs and variables encountered under clinical conditions, including thickness of zirconia and veneering ceramic, luting agent, direction, location, and type of applied load is difficult.[87]

In the present study the mean value of shear bond strength for control group was 28.30±2.92 MPa for a mean load of 454.51±4.49 N, for sandblasted specimen the mean value of shear bond strength was 33.92±2.67 MPa for a mean load of 545.52±3.99 N and that for laser treated specimen the mean value of shear bond strength was 41.67±5.16 MPa for a mean load of 666.90±8.27 N. These results were statistically analysed using Analysis of Variance (ANOVA) followed by Dunnett 't' test applied and find that these results were statistically significant ($p<0.05$) between the groups.

It the present study it was proved that surface treatment of zirconia using Neodymium-Doped Yttrium Aluminium Garnet (Nd-YAG) laser improved the bond strength between zirconia and veneering ceramic. Although surface treatment improved the bond strength of both sandblasted and laser treated zirconia; the increase in bond strength of laser treated specimen was higher than that of sandblasted one.

Scanning Electron Microscopic (SEM) study revealed that zirconia surface when sandblasted have irregular pits with consistent loss of Yttria stabilized Tetragonal Zirconia (Y-TZP) materials.

These roughened surface act as micromechanical retention for veneering ceramic and zirconia. Whereas surface treatment with Neodymium-Doped Yttrium Aluminium Garnet laser (Nd-YAG) shows somewhat regular shallow pits and flake like structures with minimal loss of Yttria stabilized Tetragonal Zirconia (Y-TZP) materials. These pits and flakes might act as micromechanical retention between zirconia and veneering ceramic. The number of pits and flakes can be controlled by adjusting the setting of laser light intensity and the depth and width of each pit can be altered by changing the duration of irradiation time.

SUMMARY AND CONCLUSION

The study was done to evaluate the shear bond strength between zirconia and veneering ceramic after surface treatment with sandblasting and laser. The zirconia block dimension was created using Design software. As per the data present from design software 30 zirconia blocks were machined to 7 mm thickness, 7 mm breadth and 15 mm length rectangular samples using milling machine and were sintered using sintering furnace. Then, these samples were cleaned ultrasonically using Ultrasonic cleaner and further cleaned with demineralized water, followed by drying in hot air.

Among thirty zirconia blocks ten were sandblasted with 110 µm alumina particles, ten were laser treated with Nd-YAG laser and the remaining ten specimens were taken as control group. All the thirty zirconia blocks were then ceramic veneered with dimensions 4 mm length 4 mm width and 4 mm thickness by conventional layering method. All these specimens undergone shear stressed in Instron 3345 Universal testing machine and the fracture values obtained were digitally recorded.

These results were statistically analysed using ANOVA (Post hoc) followed by Dunnett ‘t’ test applied and found that these results were statistically significant ($p<0.05$) between these groups.

CONCLUSION

Within the limitations of the present study, the following conclusions were drawn:

i. Scanning Electron microscopy of sandblasted specimen revealed irregular pits with considerable loss of zirconia surface material.

ii. Scanning Electron microscopy of laser pre-treated specimen revealed regular shallow pits and flake like appearance with less loss of zirconia surface material.

iii. Sandblasting the zirconia surface increased the shear bond strength between zirconia and veneering ceramic.

iv. Laser pre-treatment also increased the shear bond strength between zirconia and veneering ceramic.

v. The increase in shear bond strength between zirconia and veneering ceramic is more for laser pre-treatment when compared to sandblasting.

vi. The laser pre-treatment might be a novel technique of surface treatment for enhancing bond strength between zirconia and veneering ceramic.

FUTURE RESEARCH

In this research shear bond strength between zirconia and ceramic after surface treatment with sandblasting and laser were evaluated. This study was conducted by air abrasion with alumina particle of specific size and Nd YAG laser of specific energy setting. Sandblasting with different particle size and laser treatment with different energy settings are available. The future research on shear bond strength can be done on these variables.

Scanning Electron Microscopy (SEM) evaluated the zirconia surface after surface treatment. Scanning Electron microscopy (SEM) after shear bond strength test can be done to evaluate the type of bond failure. The future research on Scanning Electron Microscopy can be done on these variables.

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